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SAMPLING OF GLACIAL SNOW FOR PESTICIDE ANALYSIS

T. R. Stengle, et al

Massachusetts University

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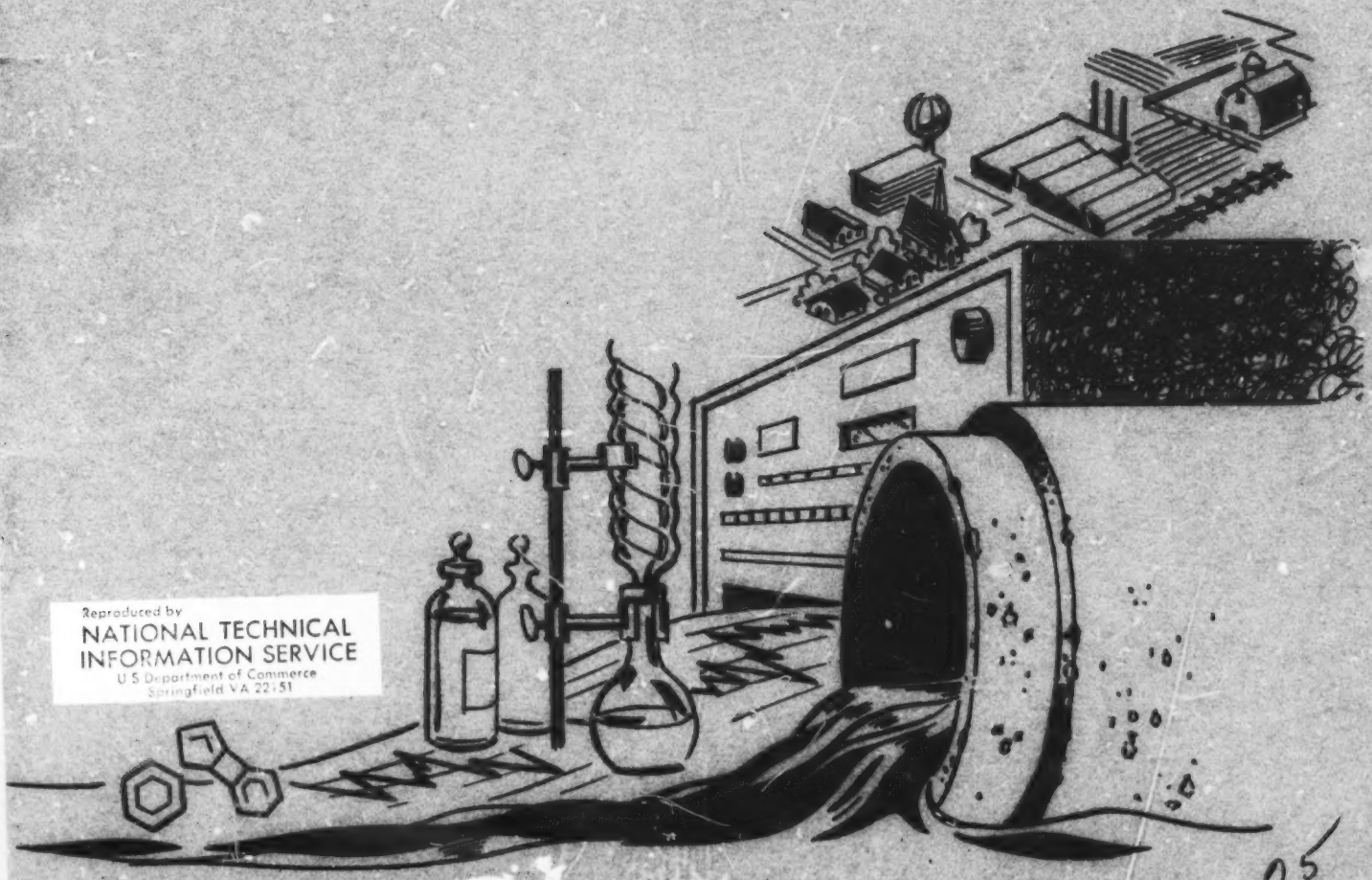

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16. Abstract

Snow samples were taken from the plateau glacier of Mt. Logan, Y. T., Canada during an investigation intent on demonstrating the feasibility of taking such samples from high altitude snowfields for pesticide analysis and developing sampling techniques devoid of contamination. Samples were also taken at a depth of 15 m to determine whether DDT concentration varied with the age of the snow. Pesticide analyses were performed using vapor phase chromatographic techniques. DDT was not detected within the limit of detectability (5 ng/l). Over half the samples were contaminated with PCB's which raised the detectability limit to 10-50 ng/l. The PCB contamination is believed to have come from the sampling auger, as an analysis of rinsings from it seemed to confirm. A sample taken from a depth of 1.5-2.5 m was acidified with distilled nitric acid, when thawed, and analyzed for 19 elements simultaneously using a direct reading emission spectrometer. Four elements were detected: boron at 4 ng/ml, cadmium at 9 ng/ml, chromium at 2 ng/ml, and iron at 47 ng/ml. The high levels of cadmium and especially iron suggest contamination from the alloy steels of the auger. (Mortland-Battelle)

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SAMPLING OF GLACIAL SNOW FOR PESTICIDE ANALYSIS

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ABSTRACT

This project was intended to demonstrate the feasibility of taking snow samples from high altitude snowfields for pesticide analysis, and to develop sampling techniques which would avoid contamination. The sampling was to be carried out in the field, under adverse conditions, with minimal equipment. Coincidentally, the DDT level in the sampling site could be determined.

The sampling was done on the plateau glacier (elevation 17,500 ft.) of Mt. Logan, Y.T., Canada. The area is remote from civilization, and the sampling team had to contend with storms, low temperatures, and the effects of high altitude. Simple techniques for obtaining samples and shipping them to the laboratory were tested. Samples were taken to a depth of 15 meters to determine whether DDT concentration varied with the age of the snow.

Analysis showed no DDT within the limit of detectability. However, over half the samples were contaminated with PCB's which raised the detectability limit to 10-50 ng/l. The limit was 5 ng/l for the uncontaminated samples. The PCB contamination is believed to have come from the sampling auger, as an analysis of rinsings from it seems to confirm. It is recommended that more attention be paid to precleaning the sampling equipment in future work.

This report was submitted in fulfillment of project # 16020 GAG under the partial sponsorship of the Water Quality Office of the Environmental Protection Agency.

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SECTION I

CONCLUSIONS

It is highly likely that the plateau glacier (elevation 17,500 ft.) on Mt. Logan, Y.T., Canada is free of contamination by DDT within the limit of detectability. The results of this study are equivocal, since some snow samples were contaminated with polychlorinated biphenyls, which mask the presence, if any, of DDT.

Samples of snow which fell many years ago can readily be obtained from glaciers, although the sampling must often be done in remote areas under adverse conditions. The samples are suitable for pesticide analysis if the sampling equipment is well cleaned in advance, and if certain precautions are followed. Sampling for trace metals is more difficult, and cannot be accomplished successfully without elaborate planning and sophisticated precedures.

SECTION VI

RECOMMENDATIONS

The results of this study were clouded because of contamination of some of the snow samples with PCB's. This presumably came from the auger used for sampling. It is recommended that this work be repeated on a limited scale with careful precleaning of the equipment in order to remove this uncertainty.

In future snow sampling the techniques developed in this work can be recommended as reliable, inexpensive, and simple to apply in the field.

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SECTION III

INTRODUCTION

Since its introduction a generation ago, enormous quantities of DDT have been used in a variety of pesticidial formulations. Fears have recently been aroused that much of the world has been polluted by residues of this non-biodegradable material. It is commonly found in surface water, and in many animals such as cattle, fish, and even humans. The extent of this contamination is not clear. It could be limited to certain well-defined pathways which lead from the original application of the material through a known chain of dilutions (with concentration points such as animal fat along the way) to eventual dissipation into concentrations too small to be detected. On the other hand the material could be so widespread that it has become a significant constituent of major portions of the earth, e.g., the atmosphere or the oceans.

Although the use of DDT as a pesticide is being phased out, the answers to such questions are important, for the DDT system can serve as a paradigm for other forms of widespread pollution, past and to come. In fact, DDT is an excellent substance for a model study. It is strictly a synthetic chemical, and there is no natural background level. Since it is not biodegradable, its presence can be detected long after it has been applied. Several laboratories have developed analytical techniques of great sensitivity for the material. Finally, as the use of DDT is phased out, its dissipation can be observed. Thus, DDT is an ideal material to study both for determining the present state of world pollution and to observe the rate of depollution as its use is discontinued.

All of the world's fresh water is ultimately traceable to the atmosphere. It is of considerable importance to determine if there are contaminants in the atmosphere which can be carried down in precipitation. The sampling of glacial snow is an intriguing approach to the problem, for it can yield data on both the present and the past condition of the atmosphere. In many glaciers snow falls from a hundred years past lie over one another; one need only bore down from the surface to sample the snow of past years. This project is an attempt to ascertain the feasibility of sampling glacial snow in the field with minimal equipment, under adverse conditions.

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SECTION IV

PREPARATIONS FOR FIELD WORK

The success of field work is entirely dependent on careful preparation. This is particularly true when the site is a glacier, for the season is short, and one is often far from civilization. It may be impossible to procure an overlooked item or repair malfunctioning equipment. In this project careful preparation was especially important, since the sampling site was very remote, and the prevailing conditions were quite hostile.

The site chosen for this work was the high plateau glacier on Mt. Logan at an elevation of 17,500 ft. For several years the Arctic Institute of North America has operated a high altitude research facility at this point during the summer season. The DDT sampling was easy to add on to that laboratory's projects, because the A.I.N.A.'s personnel have the equipment and experience necessary for obtaining snow samples at depth in glaciers.

Mt. Logan lies near the center of the St. Elias range in the Yukon Territory of Canada. It is about twenty miles east of the Alaska-Yukon boundary, and forty miles north of the Gulf of Alaska. Mt. Logan is remote from the outposts of civilization, as it is surrounded by one of the world's most heavily glaciated areas. The nearest permanent human habitation or road is nearly ninety miles away. Due to the short growing season, practically no agriculture is practiced in the Yukon, and massive applications of pesticides are unknown there.

In terms of sheer bulk, Logan is said to be the largest mountain in the world. The lower section is a massive rock, roughly ten miles long by four miles wide which culminates in a plateau at 17,500 ft. The plateau is circled by a range of seven peaks, all over 18,000 ft. The principal summit reaches to 19,850 ft. This plateau is the most favorable spot in North America for a high altitude laboratory. It is sheltered from the worst of the high mountain weather, and is readily accessible to ski-equipped aircraft. Less than an hour's flying time away, the Arctic Institute maintains a permanent base and an airstrip on the shore of Kluane Lake at 2,600 ft. elevation. This delightful spot is on the Alaska Highway just 150 miles north of the city of Whitehorse, the capital of the Yukon Territory.

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Supplies for the Mt. Logan site are trucked from Whitehorse to Kluane and then flown onto the mountain by ski plane.

For collecting the snow samples, the Arctic Institute purchased a new SIPRE auger with 30 meters of extension rods. In addition another auger was borrowed from the U.S. Army Cold Regions Research and Engineering Laboratory at Hanover, New Hampshire to serve as a backup in case the first instrument was lost or damaged. This device was designed specifically for sampling of snow and ice by the Snow, Ice, and Permafrost Research Establishment. It consists of a barrel with a bore of three inches and a length of one meter. One end is fitted with cutting blades, and the other is attached to a tee-bar handle for turning. It is constructed of stainless steel and coated with Teflon. The cutting blades are of tool steel. In use, the auger is turned by hand using the tee-bar. As the bore hole becomes deeper, extension rods are attached between the auger and the handle. These are one meter long, made of one inch aluminum pipe, and fitted at the ends with quick disconnect couplings. The extension rods are added on, one by one, as the auger is lowered into the hole, and removed again as the apparatus is raised.

By starting with a brand new auger, it seemed that any contamination of the samples from that source could be ruled out. This was not the case however, for when the apparatus was uncrated at Kluane Lake, quantities of an oily material were found on both the auger and extension rods. This was probably cutting oil which had not been removed after the machine operations. It was impossible to effect a thorough cleaning with the facilities at Kluane Lake, but most of the material was removed by swabbing the auger with paper towels soaked in Coleman fuel. This is a highly refined non-leaded gasoline designed for use in camp stoves. Even after this treatment, it was obvious that traces of the oily material remained.

Procurement of the right sample containers was an important aspect of the preparations. Two gallon wide-mouth Nalgene jugs were chosen for use on the glacier. The mouth was large enough so that the 3" snow core could be slid into the jug directly from the auger without intermediate handling. So long as the core remained frozen, it was safe to store it in plastic. However a test had shown that, given time, water would leach material (presumably plasticizer) from the jugs. Therefore, the frozen cores were flown to base camp where they were allowed to melt at room temperature: they were then transferred to glass bottler as soon as possible. These bottles have been used as water sample

containers by the WQO Analytical Control Laboratory for some time, and are satisfactory for shipping samples. A special effort was made in the precleaning of both jugs and bottles because of the low levels of pesticide expected. The cleaning process involved washing with soap and water, rinsing with tap water followed by distilled water, and finally with "distilled in glass" quality acetone. These preparations were performed by personnel of the WQO Analytical Control Laboratory in Cincinnati, Ohio. Sixteen two-gallon jugs and 60 one-quart bottles were sent to Kluane. They were not opened until used.

A number of other items not directly involved in the sampling had to be provided. A SIPRE snow kit for determining core densities was borrowed from U.S. Army CRREL. Decontamination was not a problem here, since the short core sections used for density measurement were discarded afterwards. The other items needed at the sampling site were: a tent for storage of equipment, a shovel for digging the initial pit, notebook and pencils, labels for the Nalgene jugs, and rubber bands and plastic bags. The plastic bags were used to cover the hands of the man handling the auger, and the rubber bands held them on.

SECTION V

SAMPLING

Samples could not be taken at the high altitude camp, because of the probability of contamination from generator and aircraft exhaust. Therefore, a site was selected 1,000 feet WSW (the direction of the prevailing winds) of the camp, and at a slightly higher elevation. As an additional precaution to reduce contamination, all trash at the camp was buried rather than burned. A four-man tent was set up at the sampling site to store the auger and other equipment. Next to it, a pit, one meter in depth by two meters in diameter, was dug to remove the loose overburden of recently fallen snow. The drilling took place on the floor of this pit, and the reported drilling depths are in meters below this surface. This pit also gave the drilling party some protection from the wind while working.

Precaution was taken to avoid contamination during sample collection, although the conditions on the mountain precluded the use of really sophisticated techniques. Before collecting any samples, three short holes were drilled and the cores discarded in the hope of removing any last traces of foreigh material from the auger. The drilling team was made up of three persons. Two men operated the drill and attached the extension rods, while the third worked with the auger itself. This man wore large polyethylene bags over his mittens attached by rubber bands. He alone handled the auger or touched the core. Often surgical gloves are used for such work, but their lack of warmth would have certainly resulted in frostbite. Most of the drilling was done at temperatures between 0° and -10°, and often in the wind.

The altitude of the drilling site presented a serious problem. At 17,500 ft. the barometric pressure is half that of sea level, and the effects of hypoxia are quite marked. Although the drilling team was well acclimatized to altitude, they were quickly exhausted by the heavy work. Furthermore, the lack of oxygen seems to intensify the effect of cold. The psychological effects are equally important. One's work is constantly hampered by a feeling of lassitude and an impairment of judgement.

In a typical drilling operation the auger is lowered into the hole and the extension rods are attached as it descends. The actual drilling takes only a few minutes and

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is not difficult. Lifting the auger from the hole is very hard work, and quite tiring. If one should let the auger slip when it is near the surface, and most extension rods have been removed, it would fall to the bottom of the hole. If the depth were ten meters or greater, it would be impossible to retrieve the auger, and the project would have to be terminated unless a spare auger were available.

When the auger is lifted from the hole, the person with hands bagged in polyethylene grasps it while the last extension rod is removed. Then he tips it upside down to allow any loose snow to fall out. The core is removed by tapping the auger with a piece of wood (bagged in polyethylene) while it is pointed into the mouth of a Nalgene sample jug. If the core is too long to fit into the jug, it can be broken off between the jug and auger with one man holding each. Once a team becomes familiar with the procedure, cores can be transferred routinely from auger to sample jug without touching any other surface. With each drilling about 1/3 meter of core was obtained, the rest being loose snow which was discarded.

Each sample jug held slightly less than one meter of core. The temperature on the high plateau never rises to freezing, so the samples remained frozen until they were flown off the mountain. Thus the danger of contamination by leaching of the plastic is minimized. At Kluane Lake the samples were allowed to melt at room temperature without local heating. The process took about 24 hours. As soon as it was thawed, the sample was poured into a one quart glass wide mouth bottle, and the bottle sealed. Only about one half the contents of a Nalgene jug was required to fill a one quart bottle, and the rest of the sample was discarded. In the most favorable cases the samples touched no surfaces other than the auger, the Nalgene jug, and the glass bottle. Other samples had to be touched with polyethylene bagged hands in the process of transferring it from the auger to the jug.

There are several possible sources of contamination in the sampling procedure. Despite the attempt to clean the auger, it is clear that quantities of the oily material remained on it. During the course of the drilling, bits of the Teflon coating began to flake off the auger. This material may be present in some samples, although it should have been discarded with the loose snow and not included with the core. In order to operate the drilling apparatus properly, it is necessary to cover the drilling platform with a board that has a hole large enough for the auger to pass through. Even though the board was covered with masking tape and polyethylene sheeting, splinters fell into the

drill hole. They should have been discarded with the loose snow, but it is possible that some found their way into the sample.

Several of the early cores showed a dark band on the outside about ½ inch from the cutting head of the auger. This discoloration was present only on the surface of the sample. In all cases but one, it was broken off and discarded. As a control, it was retained on sample #5. It could have been present to a lesser extent in other samples, and gone by unnoticed. In any event it had no effect on sample #5, as shown by analysis.

After the samples were thawed and placed in glass bottles, some solid material was observed in them. If a sample was shaken and held toward the light, one could see a few very small pieces of fuzz which resembled bits of wool. Neither the source nor the nature of this material has been ascertained. The drilling team wore outer clothes of nylon or nylon-cotton mix. Although they did occasionally wear wool gloves, they took great care not to touch the samples, either directly or indirectly, except with hands encased in plastic bags.

At the end of the drilling, one sample was deliberately mishandled as a control. The drilling team rubbed it with gloved hands and against their pants. If there is any contamination arising from clothing, this control should show at least 100 times as much as the other samples.

SECTION VI

ANALYSIS

As soon as possible after the termination of the Mt. Logan project, the samples contained in glass bottles were sent to the Analytical Control Laboratory, WQO, Cincinnati, Ohio for analysis. This was performed using vapor phase chromatographic techniques developed at that laboratory.

DDT was not detected in any of the samples. The lower limit of detectability for DDT was approximately 5 ng/l for eight of the samples. Due to interference, apparently from polychlorinated biphenyls (PCB's), 10 to 50 ng/l of DDT could have been present in the remaining twelve samples and not been detected. Efforts to remove the PCB's from the extract to allow for more sensitive determination of DDT were only partially successful. Thus the results are in part inconclusive. It seems highly likely however, that the DDT concentration in all samples was less than 5 ng/l on the basis of the eight samples which did not show PCB interference.

The source of the apparent PCB's (Aroclors 1260 or 1262) is not known for certain, although a residue of the oily substance on the auger is a likely suspect. There is no pattern of occurrence of the PCB's in the samples. Three scattered samples contained significant quantities of what appears to be Aroclor 1260 or 1262. Samples 1 through 3 and 5 through 10 contained varying amounts of a similar, but not identical material. The Kluane Lake sample and the five samples received in the second shipment (#15,16,17,18,20) did not contain such material. All possible sources of PCB's in the laboratory were thoroughly checked, and there is no reason to suspect that this material was introduced during the analyses.

The dark band retained in core sample 5 had no apparent effect on the analytical results. Sample 20, deliberately handled so as to contaminate it, gave a peak for p,p'-DDT on one gas chromatographic column. However, this was not confirmed by analysis on a second column.

All samples produced a broad initial peak with 3 to 4 distinct early eluting peaks which were more or less typical of the distilled water check that was made on the plastic jugs. In addition, samples 1 through 14 gave a high general background response to the electron capture detector.

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As a matter of curiosity, the pH (which may not correspond to the original pH) of randomly selected samples from the first shipment (sample 1 through 14 plus Kluane Lake) was measured on arrival in the laboratory as was that of the five samples that arrived in the second shipment. Sample 20, deliberately "contaminated" was low with pH 6.1. The Kluane Lake sample was high with pH 8.0 All others ranged from 6.3 to 6.9.

In an attempt to fix the source of the PCB contamination, the auger and one of the extension rods were sent to the laboratory where they were sampled by rinsing with hexane. The extension rod clearly contained traces of an oily material inside the stainless steel fitting at the end. This appeared to be a cutting oil. The chromatogram showed a series of early eluting peaks, but no traces of Aroclors. These early peaks would not have obscured the presence of DDT. None of the material from the rods was found in the snow samples. The rinsings from the auger gave a chromatogram which was rich in peaks, as well as a high general background response. A similar high background was observed in snow samples 1-14. This fraction also contained a quantity of halogenated material, which was an Aroclor, a mixture of Aroclors, or a similar substance. Its chromatogram resembled, but did not exactly reproduce that of Aroclors 1260 and 1262. Even though an exact match cannot be made, it is reasonable to conclude that the auger was responsible for the contamination of the snow sample. The material is a complex mixture, and it is possible that some components were preferentially lost from the auger as drilling proceeded. This contention is supported by the fact that the last five snow samples showed no PCB contamination.

One sample was obtained for trace metal analysis. It was taken as a snow core extending from a depth of 1½ to 2½ meters. It was stored in a Nalgene jug and acidified with distilled nitric acid as soon as it melted. It remained in the plastic container until use. Simultaneous analysis for 19 elements was performed on a direct reading emission spectrometer. Four elements were detected: boron at 4 ng/ml, cadmium at 9 ng/ml, chromium at 2 ng/ml, and iron at 47 ng/ml. The high levels of cadmium and especially iron suggest contamination from the alloy steels of the auger. This is not surprising; other workers have noted that it is extremely difficult to avoid metal ion contamination when steel sampling tools are used (1).

Method of Analysis (2)

Each sample was transferred to a two liter separatory funnel. The sample bottle was rinsed with 60 ml. of hexane which was then used to extract the sample. This operation was repeated a second and a third time. The combined extracts were dried by passing through a column of anhydrous sodium sulfate and collected in a Kuderna-Danish (K-D) flask. They were concentrated in the K-D to about 6 ml. on a steam bath and finally to a minimum of 2 ml. on a warm water bath. Up to 10 microliters was injected for gas chromatographic analysis on two different columns.

Gas chromatographic conditions were as follows:
1. A Microtek Model 179 equipped with a Ni^{63} detector, an aluminum column 6' x ¼" O.D. packed with Gas-Chrom Q (60/80 mesh) coated with 5% OV-17, and a nitrogen carrier flow of 100 ml./min. Temperatures were: injection port, 250° C.; column oven, 205° C.; and detector, 360° C.
2. A Perkin-Elmer Model 900 equipped with a tritium parallel plate electron capture detector, an aluminum column 8' x 1/8" O.D. packed with Gas-Chrom Q (60/80 mesh) coated with 5% QF-1 plus 3% Dow-200, and a nitrogen carrier flow of 40 ml./min. Temperatures were: injection port, 250° C.; column oven, 185° C.; and detector, 205° C.

TABLE I

COLLECTION DATA AND ANALYTICAL RESULTS FOR SNOW SAMPLES

Sample No. (Lab. No.)	Date	Depth (meters)	DDT	PCB	pH	Density (gm/cc)
1 (508)	7-13	1	ND	P	-	-
2 (509)	7-13	2	ND	P	-	0.39
3 (510)	7-13	3	ND	P	-	-
4 (511)	7-13	4	ND	+	-	-
5 (512)	7-13	5	ND	P	6.5	-
6 (513)	7-13	6	ND	P	-	-
7 (514)	7-16	6½	ND	P	-	-
8 (515)	7-16	7½	ND	P	6.4	-
9 (516)	7-16	8½	ND	P	6.3	-
10 (517)	7-16	9½	ND	P	-	0.55
11 (518)	7-20	10	ND	+	6.4	-
12 (519)	7-20	10½	ND	ND	-	-
13 (520)	7-20	11	ND	ND	6.4	-
14 (521)	7-20	11½	ND	+	-	-
15 (539)	7-20	12	ND	ND	6.9	-
16 (541)	7-20	12½	ND	ND	6.5	-
17 (540)	7-21	13	ND	ND	6.6	-
18 (542)	7-21	13½-14	ND	ND	6.4	0.54
20 (543)	8-5	1½	ND	ND	6.1	-
KLAb (506)	7-20	-	ND	ND	8.0	-
KLAa (507)	7-20	-	not analyzed		-	-

Notes:

Depths are approximate and refer to the average depth of the core.

ND - Not detected.

(+) - Aroclor 1260 or 1262

P - Similar but not identical to Aroclor 1260 or 1262.

pH values determined after arrival at Cincinnati; they may not reflect the original pH values.

Sample 20 was deliberately contaminated by handling.

Samples KLAa and KLAb were taken from the waters of Kluane Lake as a control.

TABLE II

RESULTS OF TRACE METAL ANALYSIS

Metal		
Zinc	ND	less than 2 ppb
Cadmium	9 ppb	
Arsenic	ND	less than 28 ppb
Boron	4 ppb	
Phosphorous	ND	less than 10 ppb
Iron	47 ppb	
Molybdenum	ND	less than 4 ppb
Manganese	ND	less than 1 ppb
Aluminum	ND	less than 4 ppb
Beryllium	ND	less than 0.02 ppb
Copper	ND	less than 1 ppb
Silver	ND	less than 0.2 ppb
Nickel	ND	less than 2 ppb
Cobalt	ND	less than 2 ppb
Lead	ND	less than 4 ppb
Chromium	2 ppb	
Vanadium	ND	less than 4 ppb
Barium	ND	less than 1 ppb
Strontium	ND	less than 1 ppb

Notes:

Results are for total metals (dissolved + suspended).

Results are given as nanograms per milliliter.

SECTION VII

ACKNOWLEDGMENTS

The Arctic Institute of North America provided logistic support for the Mt. Logan Project. Special recognition is given to the Institute's glacier pilot, Mr. Philip P. Upton, whose flying skill was indispensable to the existence of the high camp.

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SECTION VIII

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